

DATA VALIDATION REPORT
MICROBAC LABORATORY SDG L13101372

Project / Site Name: Environmental Remediation Services at White Sands Missile Range (WSMR), NM; CCWS-11, Open Burn/Open Detonation (OB/OD) Area

Project No.: 139791

Data Reviewer: M. Lyon, Shaw Environmental, Inc. a CB&I Company

Review Date: December 3, 2013

Matrix: Groundwater 3 field samples and 1 field duplicate

Parameters: Perchlorate 6850
Explosives 8330B
Nitrate + Nitrite, as Nitrogen 353.2

Validation Level: EPA Level III

Laboratory: Microbac Laboratories, Inc. Ohio Valley Division

Sample Delivery Group L13101372

Sample Nos.: HTA19-1013-1, HTA19-1013-2, HTA25-1013-1, and HTA20-1013-1.

Comments: Field QC sample, field duplicate HTA19-1013-2 was submitted for this SDG. Trip blank sample not applicable.

The data were reviewed and qualified according to the *Sampling and Analysis Plan/Quality Assurance Project Plan, Environmental Remediation Services, White Sands Missile Range, New Mexico October 2010; Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 2010*; laboratory-specific statistical process control criteria, and the analytical method specific requirements.

DATA VALIDATION REQUIREMENTS

Level IV or Full Validation includes all parameters listed below. Level III Cursory Validation parameters are indicated by an asterisk (*).

Organic Parameters

- * Temperature
- * Holding times
- GC/MS instrument performance check
- * Initial and continuing calibrations
- * Blanks
- * Surrogate recoveries
- * Matrix spike/matrix spike duplicate
- * Laboratory control sample / blank spike
- * Field duplicate
- * Internal standard performance
- Target compound identification
- Tentatively identified compounds
- Compound quantitation
- Reported detection limits
- System performance
- * Overall data assessment

Inorganic and General Chemistry Parameters

- * Temperature
- * Holding times
- * Initial and continuing calibration
- * Blanks
- * Matrix spike/matrix spike duplicate
- * Laboratory control sample / blank spike
- * Field duplicate
- * Matrix duplicate
- ICP interference check sample
- CVAA / GFAA quality controls
- * ICP serial dilution
- Sample results verification
- Analyte quantitation
- Reported detection limits
- * Overall data assessment

DATA VALIDATION QUALIFIER DEFINITIONS

No qualifier indicates that the data are acceptable both qualitatively and quantitatively.

- U Not detected. The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Limit of Quantitation (LOQ).
- J Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is in determinable.
- J- Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined low due to associated quality control indicators.
- J+ Estimated. The analyte was detected and positively identified. The associated numerical value is the approximate concentration of the analyte in the sample and the bias is determined high due to associated quality control indicators.
- N Tentatively identified. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
- UN Tentatively not detected, the LOQ is estimated. The analyte was analyzed for but was not detected above the reported LOQ. However, the reported LOQ is an estimate and may not be accurate or precise.
- NJ Tentatively identified. The reported concentration is an estimate. The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification and the associated numerical value represents the approximate concentration.
- R Rejected. The data are not usable. The presence or absence of the analyte cannot be confirmed.

DATA VALIDATION QUALIFIER REASON CODES

| Reason Code | Data Quality Condition Resulting in Assigned Qualification |
|--------------------------|---|
| General Use | |
| FB | Field blank contamination |
| FD | Field duplicate evaluation criteria not met |
| HT | Holding time requirement was not met |
| PR | Preservation requirements not met |
| LCS | Laboratory control sample evaluation criteria not met |
| MB | Method blank or preparation blank contamination |
| RB | Rinsate blank contamination |
| TB | Trip blank contamination |
| SDL | Sample quantitation limit exceeds decision criteria and the analyte was not detected |
| Inorganic Methods | |
| CCB | Continuing calibration blank contamination |
| CCV | Continuing calibration verification evaluation criteria not met |
| D | Laboratory duplicate precision evaluation criteria not met |
| DL | Serial dilution results did not meet evaluation criteria |
| ICS | Interference check sample evaluation criteria not met |
| ICV | Initial calibration verification evaluation criteria not met |
| MS | Matrix spike recovery outside acceptance range |
| PDS | Post-digestion spike recovery outside acceptance range |
| MSA | Method of standard additions correlation coefficient < 0.995 |
| PB | Preparation blank |
| Organic Methods | |
| CCAL | Continuing calibration evaluation criteria not met |
| ICAL | Initial calibration evaluation criteria not met |
| ID | Target compound identification criteria not met |
| IS | Internal standard evaluation criteria not met |
| MS/MSD | Matrix spike/matrix spike duplicate accuracy and/or precision criteria not met |
| SUR | Surrogate recovery outside acceptance range |
| TUNE | Instrument performance (tuning) criteria not met |
| P | The detected concentration difference between the primary and secondary column is greater than 25%. |

SAMPLE DELIVERY GROUP L13101372
LEVEL III DATA VALIDATION SUMMARY

| Analysis / Method | Temperature | Holding Times | Calibration | Blanks | Surrogate | MS/MSD | LCS | Duplicate | Other |
|-------------------------|-------------|---------------|-------------|--------|-----------|--------|-----|-----------|-------|
| Perchlorate 6850 | ✓ | ✓ | ✓ | ✓ | NA | NA | ✓ | ✓ | NA |
| Explosives 8330B | ✓ | ✓ | 7 | ✓ | 7 | NA | ✓ | ✓ | ✓ |
| Nitrate+Nitrite-N 353.2 | ✓ | ✓ | ✓ | ✓ | NA | ✓ | ✓ | ✓ | ✓ |

Notes:

✓ Indicates that all quality control criteria were met for the parameter(s)

N/A Indicates the validation criteria is not applicable to the analysis

If validation criteria were not met and the data were qualified, then details can be found at the page number indicated in the table.

DATA ASSESSMENT
PERCHLORATE (Method 6850)

I. Temperature

- A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

II. Holding Times

- A. The analysis holding times were reviewed and found to be in compliance.

III. Calibration

- A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant.

IV. Blanks

- A. Method blank and CCB analysis results were reviewed and found to be in compliance.

V. MS/MSD

- A. MS/MSD analyses were not reported for this SDG. MS/MSD were evidenced on the instrument run log but were not identified as a WSMR sample.

VI. LCS

- A. The LCS results were reviewed and found to be in compliance.

VII. Duplicate

- A. Field sample HTA19-0212-1 and field duplicate sample HTA19-0212-2 results were compared and the precision results were acceptable. Additionally the laboratory analyzed a LCS duplicate in lieu of the MS/MSD. LCS/LCSD precision result met specifications.

VIII. Other

- A. Samples were analyzed at 5,000-times and 10,000-times dilutions due to high concentrations of the target analyte.

EXPLOSIVES (Method 8330B)

I. Temperature

- A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

II. Holding Times

- A. The analysis holding times were reviewed and found to be in compliance.

III. Calibration

- A. Calibration coefficient of determination, alternate source calibration verification, and CCV, were reviewed and found compliant with exceptions. Percent recoveries for the compound tetryl failed criteria in alternate source calibration check on the instrument designated HPLC4. The only applicable analysis results from instrument HPLC4 was a 10-times dilution confirmation analysis for RDX in sample HTA20-1013-1. Tetryl from the 10-times dilution analysis is qualified estimated non-detect with “UN” and is not reportable. All other analysis results were from instrument HPLC5 which met all calibration check criteria..

IV. Blanks

- A. Method blank and CCB analysis results were reviewed and found to be in compliance.

V. MS/MSD

- A. MS/MSD analyses were not reported for this SDG.

VI. LCS

- A. The LCS results were reviewed and found to be in compliance.

VII. Duplicate

- A. Field sample HTA19-1013-1 and field duplicate sample HTA19-1013-2 were submitted for analysis in this SDG. All analysis results were non-detect. Precision results for the LCS duplicate were acceptable.

VIII. Other

- A. Surrogate spike recoveries were compliant with one exception. In the undiluted confirmation analysis of HTA20-1013-1 the surrogate 1,2-Dinitrobenzene recovered high, greater than the acceptance limit. None of the confirmation analysis results are reportable. Confirmation for HMX was acceptable and confirmation for RDX in a 10-times dilution analysis was acceptable and reportable.

NITRATE + NITRITE AS NITROGEN (Method 353.2)

I. Temperature

- A. Shipping cooler temperature was measured at 2 °C upon receipt at the laboratory. Sample temperature was in compliance.

II. Holding Times

- A. The analysis holding times were reviewed and found to be in compliance.

III. Calibration

- A. Calibration coefficient of correlation was reviewed and found compliant.

IV. Blanks

- A. Method blank was reviewed and found to be in compliance.

V. MS/MSD

- A. MS/MSD analyses were not reported for this SDG. MS/MSD performed in the analytical batch was on a non-project sample.

VI. LCS

- A. The LCS results were reviewed and found to be in compliance

VII. Duplicate

- A. Field sample HTA19-1013-1 and field duplicate sample HTA19-1013-2 results were compared and the precision results were compliant. A LCS duplicate was also analyzed. The results were reviewed and found to be compliant.

VIII. Other

- A. LOQ was reviewed and found compliant.

DATA QUALIFICATION SUMMARY

CCWS-11, Open Burn/Open Detonation (OB/OD) Area, four samples; HTA19-1013-1, HTA19-1013-2 (field duplicate), HTA25-1013-1, and HTA20-1013-1.

Perchlorate – Data Qualification Summary

No sample data were qualified in this SDG.

Explosives – Data Qualification Summary

No reportable sample data were qualified in this SDG. A low recovery for tetryl in an alternate source check (initial calibration check) was noted and the tetryl result qualified “UN” for non-detect and estimated in one applicable analysis which was not reportable. One surrogate failed high, greater than acceptance limits but in an undiluted confirmation analysis which was not reportable.

Nitrate plus nitrite – Data Qualification Summary

No sample data were qualified in this SDG.

OVERALL ASSESSMENT OF DATA

I. Compliance with method and project requirements

- A. All analyses were performed within the analytical methods specifications and project requirements.

II. Usability

- A. Based on the quality control criteria reviewed, all unqualified data are usable for project purposes. No reportable data results were qualified during data validation. No data results were rejected as unusable. Data qualifiers assigned by the laboratory in the analytical report include the “J” qualifier when analytes were identified but at concentrations less than the LOQ. Estimated results are usable for limited purposes.

III. Duplicate Sample Precision

| Method | Parameter | Parent Sample HTA19-1013-1 | Qual | Duplicate Sample HTA19-1013-2 | Qual | Units | RPD |
|--------|----------------------------|-------------------------------|------|----------------------------------|------|-------|-------|
| 6850 | Perchlorate | 9350 | | 9300 | | ug/L | 0.5% |
| 8330B | 1,3,5-Trinitrobenzene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 1,3-Dinitrobenzene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 2,4,6-Trinitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 2,4-Dinitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 2,6-Dinitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 2-Amino-4,6-dinitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 2-Nitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 3-Nitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 4-Nitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | 4-Amino-2,6-dinitrotoluene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | HMX | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | Nitrobenzene | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | RDX | 1.11 | U | 1.10 | U | ug/L | NC |
| 8330B | Tetryl | 1.11 | U | 1.10 | U | ug/L | NC |
| 353.2 | Nitrate-Nitrite (as N) | 21.1 | | 25.1 | | mg/L | 17.3% |